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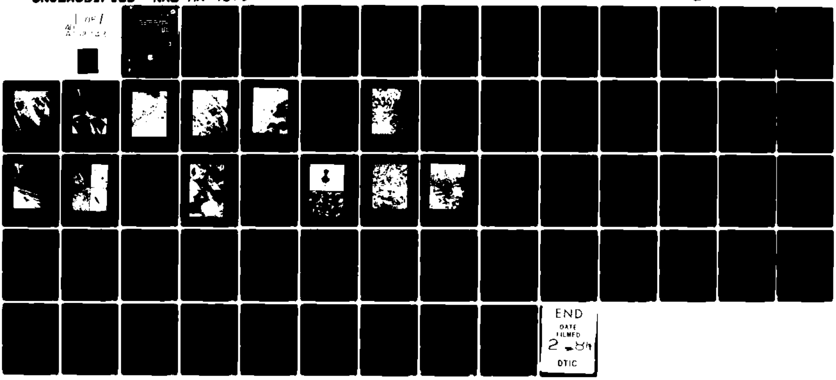

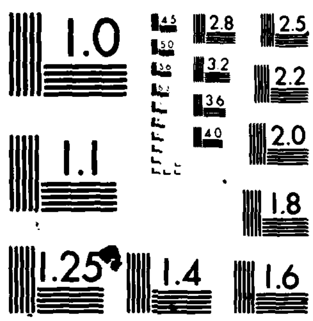

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The Cooperative Radiation Effects Simulation Program (CORES) is a collaborative effort of the Material Science and Technology and the Radiation Technology Divisions of the NRL Material Science and Component Technology Area. The goal of the research is to provide the theoretical and experimental bases for understanding the mechanisms of nuclear radiation damage of metals, as well as a theoretical insight into energy deposition processes. In this the Van de Graaff and Cyclotron are used to simulate rapidly the radiation damage produced over long periods in reactor neutron environments.

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19. Key Words (Continued)

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Transport theory
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Voids
Void growth

20. Abstract (Continued)

Progress for the period, 1 September 1978 to 31 August 1979, includes: (1) the effect of Ni^+ ion bombardment on nickel and binary nickel alloys was investigated at 675°C, 625°C and 525°C and compared with neutron irradiation at 455°C; (2) the microstructures of titanium scoping alloys following low fluence neutron irradiation at 450°C were studied by transmission electron microscopy; and (3) X-ray diffraction measurements of early stages of radiation damage in metals was studied in single crystal and polycrystalline Cu specimens.

CONTENTS

COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Annual Progress Report
for the Period
1 September 1978 - 31 August 1979

Program Description

The Cooperative Radiation Effects Simulation Program (CORES) was initiated voluntarily by five Branches from the Engineering Materials (now the Material Science and Technology Division) and the Radiation Technology Division of NRL on the basis of their common interests in the problems of simulating radiation damage in metals. The program promotes the exchange of information, discussion of problems, and the pursuit of collaborative research efforts. Annually a written report is prepared containing those portions of the work of the participating Branches which are judged to be of interest to the damage simulation problem. The major portion of the work is sponsored by the Office of Naval Research. Since research findings which apply to the objectives of one sponsor may also be of interest to others, the overall progress related to damage simulation is included in the written report. Several of the participating Branches have independent programs on other aspects of the radiation damage problem; when results obtained in these programs are judged to be of interest to CORES participants they may also be included, informally, in the CORES program review.

COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Annual Progress Report
for the Period
1 September 1978 - 31 August 1979

SUMMARY

I. HEAVY ION AND NEUTRON DAMAGE STUDIES

A. Effect of Ni^+ Ion Bombardment on Nickel and Binary Nickel Alloys

Pure nickel and four binary nickel alloys have been subjected to high energy Ni ion bombardment at 675°C, 625°C and 525°C. After irradiation, each specimen was studied by transmission electron microscopy. The pure nickel control was found to swell appreciably (1 to 5%) and the Ni-Al and Ni-Ti samples were found to swell at all temperatures, but to a lesser degree (0.01 to 0.35%). The Ni-Mo contained a significant density of voids only at 525°C, while swelling was suppressed at all temperatures in the Ni-Si alloy. The dislocation structure progressed from loops to tangles as temperature increased in all materials except the Ni-Ti, in which there was an absence of loops at all temperatures. Dislocation densities increased as temperature increased in all samples. These results do not correlate well with the relative behavior of the same alloys observed after neutron irradiation at 455°C. The differences between these two sets of data appear to be caused by different mechanisms controlling void nucleation in ion and neutron irradiation of these alloys.

B. The Microstructures of Titanium Scoping Alloys Following Low Fluence Neutron Irradiation at 450°C.

The titanium scoping alloys Ti-6Al-4V (beta annealed), Ti-6242S, Ti-38-6-44, and Ti-15-333 were examined by transmission electron microscopy (TEM) following irradiation in EBR-II to a fluence of 3.4×10^{21} neutrons/cm^2, E > 0.1 MeV, equivalent to 2.1 displacements per atom (dpa), at a temperature of 450°C. The principal microstructural changes observed in the Ti-6Al-4V were the formation of small dislocation loops and some nearly planar features identified as beta-phase precipitates in the alpha grains of the alloy. In the Ti-6242S, the major irradiation-induced microstructural change was a dense distribution of small dislocation loops in the primary alpha grains. Electron diffraction patterns indicated the probable presence of alpha -2, Ti_3(Al,Sn) but this phase could have precipitated either during heat treatment or during irradiation. The Ti-38-6-44 and Ti-15-333, which are metastable beta alloys containing alpha-phase precipitates, both precipitated additional alpha phase during during irradiation. The precipitation in the metastable beta alloys was consistent with that which occurs in these alloys during long thermal aging at the irradiation temperature, although the irradiation probably enhanced the transformation kinetics. The precipitation of fine beta phase in the Ti-6Al-4V, however, appeared to be an irradiation-induced effect, since the known thermal phase diagram for the alloy indicates that the pre-irradiation alpha phase should be stable at the irradiation temperature.

II. LIGHT ION BEAM STUDIES

A. X-Ray Diffraction Measurements of Low-Level Radiation Damage

X-ray diffraction gives a quantitative measure of early stages of radiation damage in metals. Previous work on single crystal pure Cu has been extended to higher levels of damage. A related experimental method using polycrystalline specimens has been developed because polycrystalline specimens are easier to make and provide more information. A model of the effect of radiation damage on the diffracted intensity has been developed where the observed intensity increases are attributed to internal strain (lattice spacing variation) caused by irradiation while observed intensity decreases are attributed to dislocation loops.

COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Annual Progress Report
for the Period
1 September 1978 - 31 August 1979

INTRODUCTION

This report summarizes annual research accomplishments of the interdivisional cooperative program in its ninth year. The goal of this program has been to use ion simulation techniques to increase the understanding of neutron damage of materials for advanced nuclear systems. Light and heavy ion bombardment techniques are used to represent neutron damage in order to accelerate research and to permit an evaluation of critical parameters which might not be feasible using nuclear reactors as test facilities. In this program the techniques applied have advanced along with our growing understanding of radiation damage of materials. To complement the ion damage simulation are other programs such as theoretical evaluations of atomic collisions, computation of energy deposition, and parallel experiments using nuclear reactors.

The accomplishments of the CORES Program have been numerous and impressive as documented by the full list of technical papers and presentations in the last section. Recognition has come to NRL as personnel have been asked to contribute in the development of nuclear technology in the USA by service on panels, committees, workshops, conferences and task forces. Many new spin-off programs such as ion-implantation for materials modification, energy transport codes for ion-materials interactions and other investigations in radiation effects in materials, germinated and grew from the CORES Program. In this final CORES Progress Report it is fitting to list some significant accomplishments of the program:

1. The development of the E-DEP-1 Code to calculate the number of atoms displaced by charged particle bombardments.

2. The development of a versatile, high current density heavy ion source and capabilities for performing high temperature bombardment.

3. The development of quantitative electron microscopy techniques to characterize swelling resistance of materials from charged particle irradiations.

4. The development of a versatile apparatus to measure steady state and transient irradiation creep rates during deuteron and alpha bombardment.

5. The development of the alpha-alpha scattering technique to quantitatively measure helium concentration profiles in thin metal foils.

Manuscript submitted September 4, 1980.

6. The development of a capability to implant high concentrations of helium into test specimens at controlled temperatures.

7. The performance of a series of experiments which established the influence of dose rate on swelling.

8. The performance of a comprehensive series of experiments that established the temperature dependence of swelling in nickel and related it to microstructural changes occurring in the material.

9. An investigation of the effects of alloy additions on swelling behavior of nickel.

10. A quantitative comparison of swelling produced by ion bombardment and neutron irradiation which showed the disparities in behavior.

11. An investigation of precipitate stability under ion irradiation in Ni-Al alloys which documented the major effects.

12. A study of the mobility of helium in solids which showed the mechanism to be random motion and collision of bubbles.

13. A characterization of the effect of helium on the fatigue behavior of 316 stainless steel and the molybdenum alloy, TZM.

14. A study of the mechanisms of transient and steady state creep of Ni during deuteron and alpha bombardment which showed climb-controlled glide to be dominant.

15. A critical assessment of the ability to simulate the swelling produced by neutron irradiation with accelerated rate ion bombardment.

16. Critical testing of void nucleation and growth models and theories of swelling with a comprehensive set of experiments on a pure material, Ni, and carefully controlled irradiation conditions.

17. Fostering educational development in this area through support of PhD thesis work of Peter Hendricks at MIT and George Kirchner at the University of Wisconsin, and M.Sc. thesis work of Karen Roarty at the University of Virginia.

RESEARCH PROGRESS

I. HEAVY ION AND NEUTRON DAMAGE STUDIES

A. Effect of Ni^+ Ion Bombardment on Nickel and Binary Nickel Alloys

(K.B. Roarty, J.A. Sprague, F.A. Smidt, Jr., Naval Research Laboratory, and R. A. Johnson, University of Virginia)

Introduction

Void formation in cladding and structural alloys is a major materials problem for fast breeder and fusion power reactors. A large number of fast reactor experiments have been conducted to determine the effects of temperature and dose upon the amount of swelling in various materials [1-3]. However, the time required for many materials to receive a sufficient dose to yield significant swelling is on the order of one to several years, making the gathering and analysis of data difficult. In order to expedite the research on void formation, high energy charged-particle bombardment has been used to simulate fast neutron damage with a resultant three-to-four orders of magnitude increase in the displacement damage rate. Although the quantitative correlation of charged-particle and fast neutron irradiations has proven to be an elusive goal, [4] charged-particle experiments have provided very useful information on the mechanisms of void formation and on the relative swelling rates of different alloys.

The present study was undertaken to expand on a previously-reported scoping experiment [5] to examine the effects of individual solid-solution alloying elements on void nucleation and growth in nickel by Ni-ion irradiation. The results of these experiments were compared with preliminary results of a companion study involving neutron irradiation of the same alloys and with data reported previously by other investigators for Ni-ion irradiation of a similar set of nickel alloys. The comparisons provide some insights into the mechanisms of alloying effects on swelling and into the use of ion irradiation as a tool for screening the relative swelling of various alloys.

Description

Four binary nickel alloys of 1 at.% aluminum, titanium, silicon and molybdenum and a pure nickel control were prepared by arc melting in a titanium-gettered argon atmosphere. A 3-mm thick slice was cut from the center of each arc-melt button and rolled to 0.1-mm thick foil from which 3-mm diameter disks were punched. The Ni-Al alloy was annealed for one hour at 800°C to avoid evaporation of aluminum, while the other samples were annealed for one hour at 990°C. The materials were then mechanically polished and reannealed at 800°C for two hours to remove defects induced by handling and polishing. All anneals were carried out in a vacuum of 1 x 10^{-5} Pa. Finally, the surface of each specimen was lightly electro-polished in a solution of 250 ml methyl alcohol, 150 ml butyl alcohol, and 10 ml perchloric acid at -65°C to remove the polishing scratches and the grain relief resulting from the vacuum anneal.

The ion irradiation, specimen thinning, and TEM observation procedures have been described in detail in a previous publication, [6] and will be summarized here. The specimens were bombarded with 2.8 MeV Ni^{+} ions in the Van de Graaff facility at the Naval Research Laboratory. The experiments were conducted at four temperatures, 675, 625, 575 and 525°C. Each specimen was irradiated with a beam current density of 5 to 6$\mu A/cm^2$ to a fluence of 5.68 x 10^{15} ions/cm^2. Using the E-DEP-1 computer code [7] and assuming a displacement energy of 40 eV, the peak dose was calculated to be 8.1 dpa with a damage rate of 4 x 10^{-2} dpa/sec.

Since the maximum damage region is predicted by E-DEP-1 to be between 4500 and 5000 Å, the specimens were front-face polished using an interferometric electropolisher [8] to a depth of slightly less than 5000 Å. To obtain electron transparent areas of the now-exposed damaged regions, the specimens were backthinned in a dual-jet electropolisher with the same electrolyte and polishing conditions used for the final pre-irradiation surface preparation. The polished, irradiated face was protected by a layer of petroleum jelly and a plastic film. Unfortunately, due to unsatisfactory front-surface electropolishing, the specimens irradiated at 575°C could not be analyzed, so that only data from 525, 625 and 675°C could be reported.

The materials were analyzed using a JEOL-200-A electron microscope at the peak operating voltage of 200 kilovolts., By means of tilting the specimen stage, an area was photographed with an angular difference of between 10 and 20 degrees and these micrographs were then studied in a stereoviewer to determine the thickness of the area. Void number and size were measured using a Zeiss particle size analyzer.

Experimental Results

Void and dislocation data for the five ion-irradiated materials are summarized in Table 1. The table also includes preliminary void data from a comparison study of neutron irradiation of the same materials in the EBR-II reactor [9]. The irradiation conditions for these specimens were 2.2 x 10^{22} n/cm^2, E > 0.1 MeV, (7.6 dpa) at 455°C. More detailed analyses of the neutron irradiated specimens will be published elsewhere [10].

The general characteristics of the void microstructure observed in the pure nickel were high void densities and wide void size distributions, as shown in Fig. 1. The swelling from these voids increased monotonically with increasing temperature over the range studied.

The void density was much smaller in the Ni-Al material than in the pure nickel control, while the mean void diameter was similar, yielding a net decrease in swelling of about two orders of magnitude. Also, the void sizes were much more homogeneous in the Ni-Al alloy than in the pure nickel control (Fig. 2). An area of dense voids was found in the Ni-Al material at 675°C (Fig. 3). This dense band of voids represented less than 1% of the specimen, but the density and swelling were an order of magnitude greater than any other area of the specimen. This phenomenon was regarded as due to a heterogeneity of the sample, rather than as an effect of the irradiation.

The Ni-Ti material also suppressed swelling but to a lesser degree than the Ni-Al material. Again, the resulting decrease in swelling, in comparison to the pure nickel control, was due to a decrease in the void density as the mean void diameters were about the same as their pure nickel counterparts. The decrease in swelling between the Ni-Ti material and the pure nickel control was about an order of magnitude.

As in Ni-Al, the void size of the Ni-Ti was fairly homogeneous with void diameter increasing with increasing temperature. However, a pattern of void nucleation was observed in all the Ni-Ti specimens that was not observed in any other material. Throughout most of the area of each specimen, the voids appeared to be scattered randomly. Yet in each of the Ni-Ti specimens, in addition to the random scattering, well-defined rows or "strings" of voids also appeared (Fig. 4). The length of these strings varied from area to area and by means of examining stereo pairs, one could see that some of them travelled from one surface of the specimen to the other. Frequency of the strings increased with the increasing void density at the lower temperatures. Further examination of the material, by means of a series of micrographs taken of the same area at different tilt angles, revealed that voids were growing along dislocations. Since the void strings were prevalent in only one material, it is thought that they are the result of voids nucleating and growing along dislocations present in the material prior to irradiation.

Another interesting void formation occurred at the lowest temperature, in which voids grew and clustered together to form dendritic voids (Fig. 5). A dendritic void consists of a central void with other voids attached to each of its six truncated faces. Dendritic voids, both symmetric and non-symmetric, have been seen by other researchers in similar materials [11,12]. A non-symmetric dendritic void is one with either fewer than six appendages or with dissimilar appendages.

In contrast to both the Ni-Al and the Ni-Ti materials, the Ni-Mo alloy suppressed swelling drastically. Only at 525° were voids of any

Fig. 1 — Pure nickel control at 625 C. Note the heterogeneity of void size and density, particularly the denuded zones on either side of the grain boundary

Fig. 2 — Ni-1% Al at 675 C. This area is typical in both void size and density

Fig. 3 — Ni-1% Al at 675 C. This is the same sample as in Fig. 2, but a different area, showing a band of very dense, heterogeneous voids

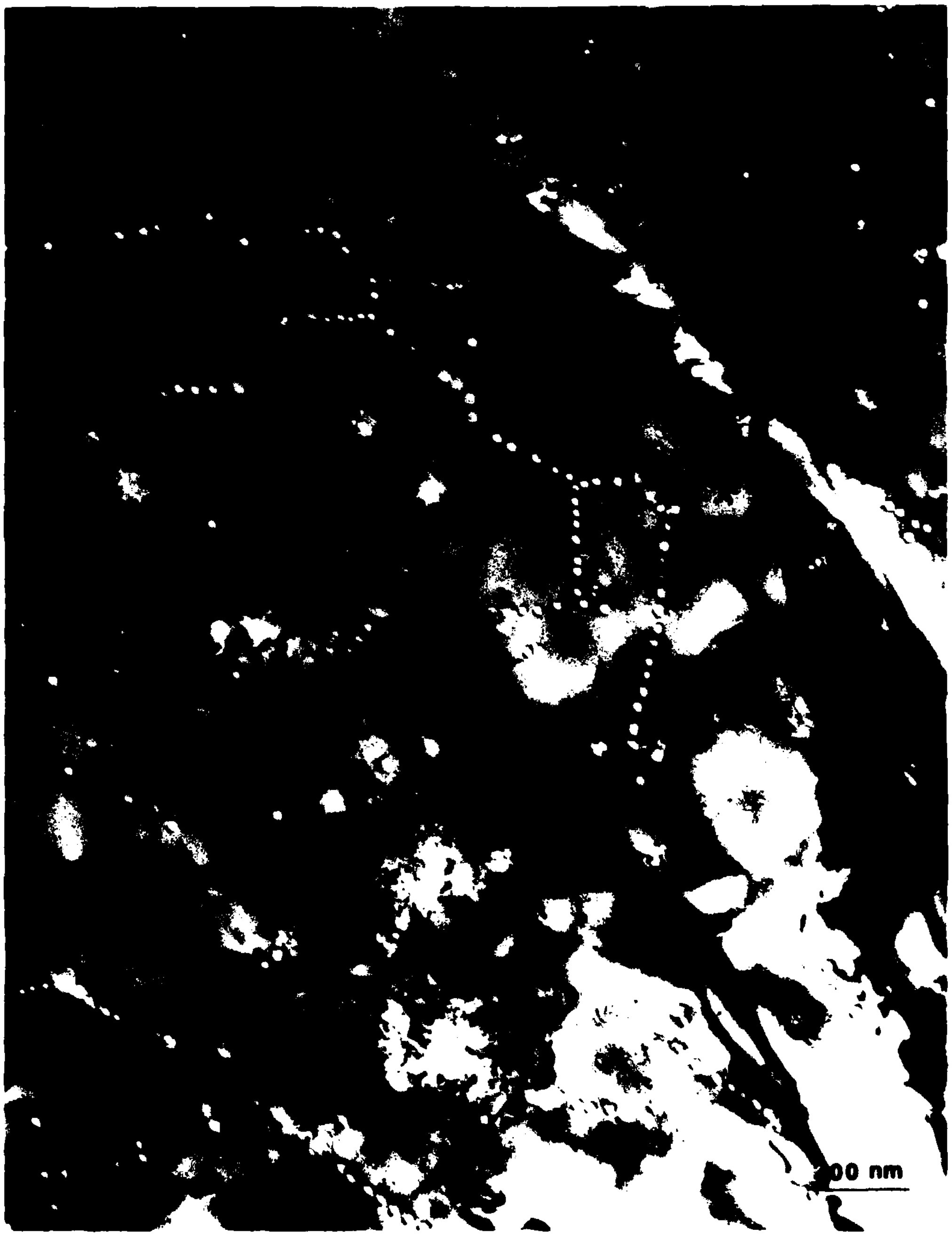

Fig. 4 — Ni-1% Ti at 525 C. Shown here are the strings of voids typical of the Ni-Ti alloy

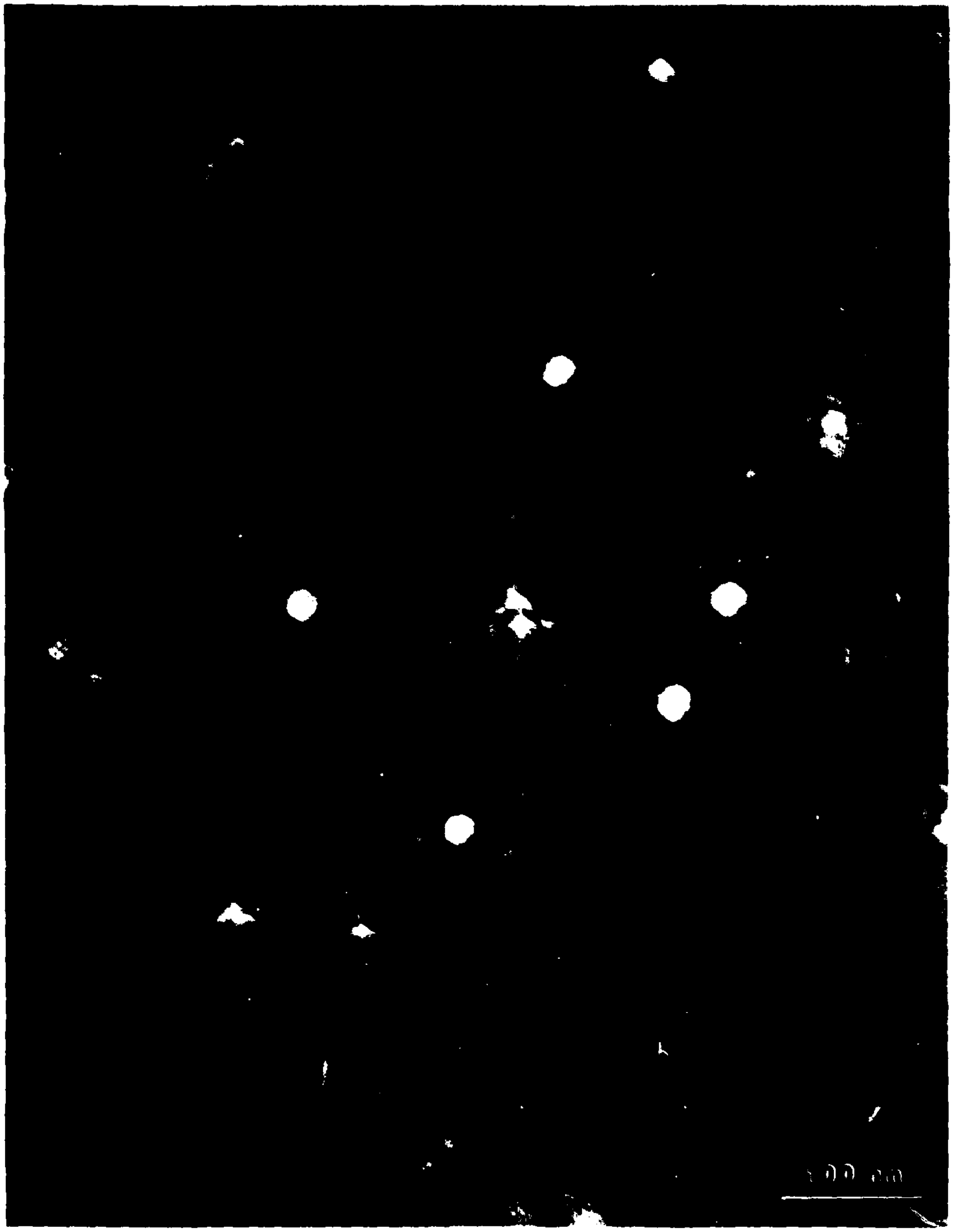

Fig. 5 — Ni-1% Ti at 525 C. In the center of the picture is a dendritic void that is entirely within the foil. Note that the size of each of the non-dendritic voids surrounding it is about the same as the size of each of the appendages. The large dendritics at the bottom of the picture are intersecting the surface.

consequence seen. These were scattered fairly evenly throughout the sample although the density was very low.

Of all the materials examined,, the Ni-Si suppressed swelling the most effectively. There was no void formation seen at any temperature, thus suggesting a complete suppression of void nucleation (Fig. 6).

In most of the materials examined, the dislocation density decreased with increasing temperature and the dislocation structure followed a regular pattern with changing temperature. At the lower temperatures, loops were prevalent. As the temperature increased, the loop structure gave way to tangles of varying density. This pattern was absent in the Ni-Ti material, where the dislocation density was roughly the same at all temperatures as was the structure of tangles with a noted absence of loops.

Discussion

Based on the findings of several researchers, it has been determined that metals subjected to fast neutron or charged particle bombardment will react in a certain manner to changes in irradiation conditions. First of all, as temperature of irradiation increases above the threshold temperature for void formation, void number density increases very rapidly from zero and then decreases with increasing temperature [6,13]. In the void formation regime, void diameter generally increases with increasing temperature. Furthermore, dislocation density increases with increasing temperature until void nucleation and growth begin to dominate, at which point the dislocation density begins to decrease [14].

The general relationship between void growth and dislocation density can be seen theoretically as follows. At low temperatures the mobility of the vacancies is low and the dislocations are nucleating in the form of small interstitial loops. As the temperature of irradiation is increased, the mobility of the interstitials and vacancies is increased as well. Since interstitials are preferentially attracted to the dislocations and dislocation loops due to the nature of the strain fields, voids, which have a less strong preferential attraction for interstitials, can nucleate and grow. This basic scenario has been the subject of considerable theoretical elaboration by several authors, involving point defect trapping, dissolved gases, applied stress and other effects (see, for example, Mansur [15] and Russell [16]).

In examining the data, it is seen that the temperature dependences of void and dislocation parameters were generally similar to those observed previously in many metals and alloys, but that there are some significant differences worthy of comment. Comparing the pure nickel results with those obtained for identical irradiation* of another batch of nickel, [6]

*In the original reference, the damage level was quoted as 13 dpa, based on a displacement energy of 25 eV. A displacement damage of 40 eV was used in the present work to conform to current convention.

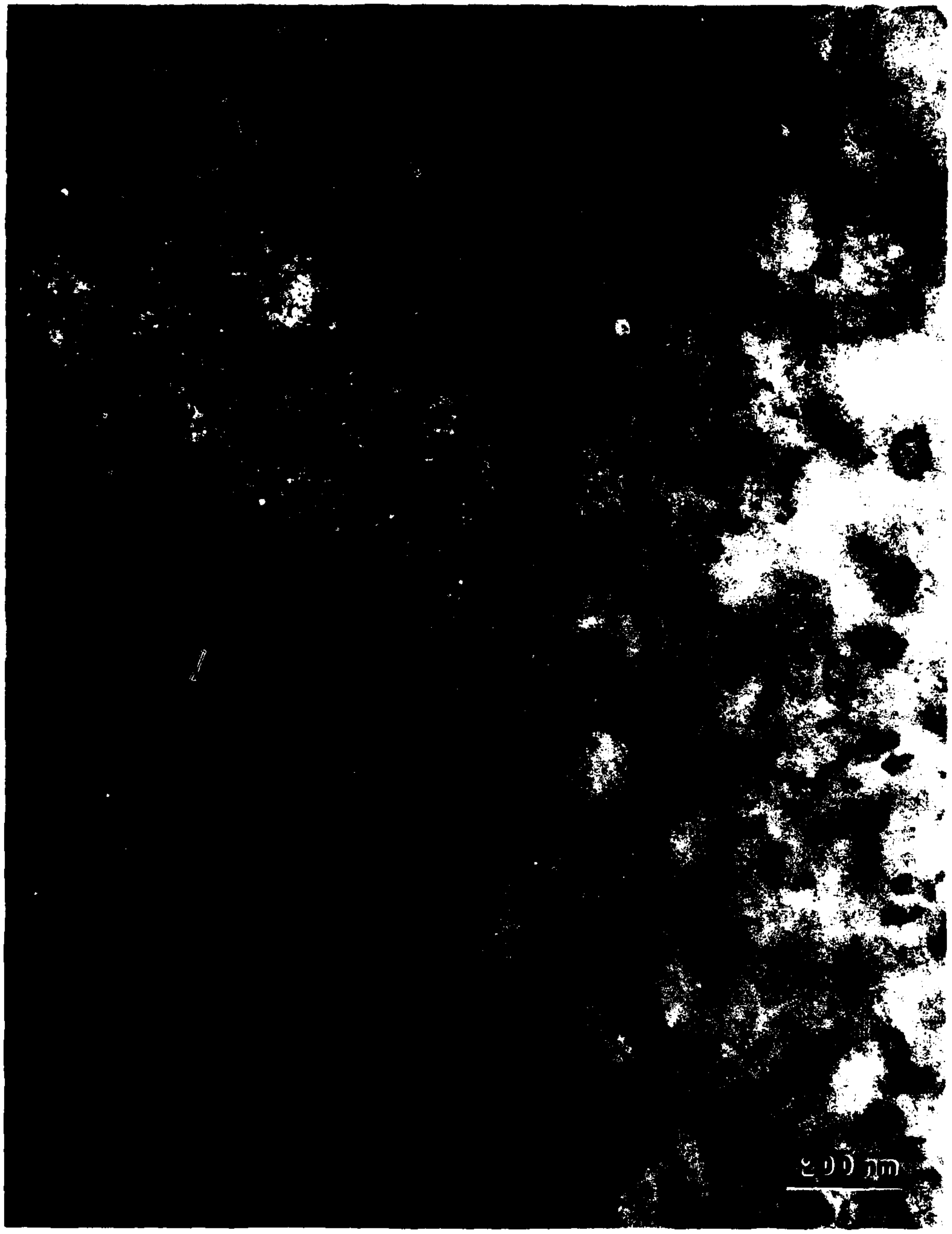

Fig. 6 — Ni-1% Si at 625 C. Typical area showing dislocation structure and density

several differences are apparent. The magnitudes of swelling were considerably larger, and the temperature dependence of the void number density was much flatter (nearly constant, within a ±30% error estimate) in the present experiment. The temperature of maximum swelling was 675°C, as opposed to 625°C in the previous work. The high swelling at 675°C was accompanied by a higher (5X) dislocation density. A possible explanation for these differences is that the present specimens, due either to higher impurity content or a subtle difference in surface preparation, retained more of the irradiation-produced dislocations, providing the necessary biased sink for void nucleation and growth at higher temperatures.

Looking at the alloying effects in the present experiment, the swelling suppression produced by all four elements was due to a decrease in void number density, implying a strong effect of alloying on void nucleation. This type of effect is not too surprising, since theories of homogeneous void nucleation, [17,18] predict a great sensitivity of nucleation rate to reductions of vacancy supersaturation, which could be produced by vacancy or interstitial trapping [15,19]. Another interesting point is that the alloy with the greatest swelling, Ni-Ti, also showed a retention of dislocation density to high temperatures, while the Ni-Al, Ni-Mo, and Ni-Si all showed fairly pronounced decreases in dislocation density with increasing temperature.

A similar experiment performed by Potter et al. [20] using similar alloys and a lower ion dose, yielded different results. Void densities and swelling were greater than those in the present experiment. The Ni-Al and the Ni-Ti alloys suppressed swelling as in the present experiment, but the Ni-Mo alloy suppressed swelling less effectively and the Ni-Si alloy contained voids at three of the four temperatures, whereas in the present experiment, no voids were found at any temperature. Also, they found some strings of voids, such as reported for Ni-Ti in the present experiment, but only for Ni-Mo [21].

Potter, et al. correlate the ability to suppress swelling as a function of solute atom misfit. The undersize misfits, such as the silicon, should suppress swelling the most effectively, as is indicated by both experiments. In the case of the oversize misfits, the aluminum, titanium and molybdenum, the ability to suppress swelling should increase with increasing misfit size, which occurred in Potter's experiment, but not in the present investigation.

Differences in data from the two experiments notwithstanding, the one outstanding contradiction is in the theoretical prediction that swelling should increase with increasing dose. Potter's experiment, performed at a lower dose than the present experiment, had consistently higher amounts of swelling. These differences in data may be due to a strong effect of impurity content or specimen preparation on void nucleation.

There is disagreement among researchers [22] as to what method of analysis provides the most reliable data in an ion irradiation. The same materials, subjected to the same Ni^+ ion bombardment at three different sites, provided different results at each site, indicating the variance possible due to slightly different irradiation conditions. There is also disagreement as to the role of ion irradiation, or any charged particle bombardment, in predicting the behavior of a material subjected to high fluence neutron irradiation. Due to a lack of neutron data on materials which have been subjected to ion irradiation, there is little concrete evidence with which to formulate correlations between the neutron irradiations and the simulations. Garner, et al. [23] propose that due to the basic differences in properties between neutron and ion irradiations, it is doubtful that any simulation would predict the behavior of a material subjected to high fluence neutron irradiation. Rather, the simulation should be used as a screening device for potential alloys for neutron irradiation.

Preliminary data, however, of such a neutron irradiation are now available. Sprague, et al. [9] analyzed the same alloys examined in the present experiment after irradiation in the EBR-II reactor to a fluence of 2.2×10^{22} neutrons/cm^2 at 455°C. Examination of the data from this experiment is still in progress, but preliminary data indicate that there are problems with the ion irradiation as a simulation in these alloys. The pure nickel control swelled appreciably as in the ion irradiation, but with a lower density and a larger void diameter. The greatest difference occurred in the Ni-Al, Ni-Mo and Ni-Ti alloys. All were found to have greater swelling than the pure Ni, with greater void densities occurring in the Ni-Mo alloy and larger or similar void diameters occurring in all three alloys. Only the Ni-Si alloy indicates a correlation between the two experiments. Swelling was suppressed in the neutron environment, but by a different mechanism than in the ion experiment. Voids were present at similar densities in the Ni and Ni-Si in the neutron experiment, but the voids in Ni-Si were smaller, so that the mechanism of suppression of swelling was the suppression of void growth rather than void nucleation. Regarding the behavior of the alloys in the ion irradiation, it seems that although suppression of both nucleation and growth may be occurring, suppression of nucleation is more responsible for the reduction of swelling.

The most likely explanation for the observed differences in alloying effects between the ion and neutron experiments is that different mechanisms control void nucleation in the two types of irradiation. The high dose rate of an ion irradiation produces high point defect supersaturations, which provides sufficient driving force to nucleate voids without the participation of insoluble gases, such as helium. This nucleation mechanism, however, is sensitive to alloying effects which slightly alter the point defect balances. Under neutron irradiation, the lower displacement rate produces lower point defect supersaturations, but the neutrons also produce helium by transmutation reactions, which can drive void nucleation at the lower supersaturations. It seems reasonable that this gas-driven nucleation mechanism would be less sensitive to low-concentration alloying effects such as point defect trapping. A parametric study using a general void nucleation theory, [16] would be required, however, to test this hypothesis.

Summary and Conclusions

Pure nickel and four binary nickel alloys containing 1 at.% of aluminum, titanium, molybdenum, and silicon respectively, were examined by transmission electron microscopy following irradiation with 2.8 MeV Ni^+ ions to a peak damage level of 8.1 dpa at 525, 625, and 675°C. The maximum swelling observed in pure nickel was 4.1% at 675°C. The maximum swelling was suppressed by a factor of 10 by the titanium addition, a factor of 100 by the aluminum, a factor of 1000 by the molybdenum, and completely by the silicon. The swelling suppression was by a reduction in void density.

Comparing the present results for pure nickel with a previous experiment involving identical ion irradiations of a higher purity batch of nickel, the peak void swelling in the present experiment was nearly a factor of four higher and occurred at a higher temperature. The difference in high temperature swelling behavior appeared to be related to a greater dislocation density retained at high temperature in the present material.

The present results agree with a previous ion-irradiation experiment by Potter, et al. [20] with regard to the effect of silicon, but do not agree with regard to the effects of aluminum, titanium, and molybdenum, which Potter, et al., found to be much less effective in suppressing void nucleation.

In comparing the results of the heavy ion bombardment with the preliminary results of the fast neutron irradiation, it seems that different mechanisms were controlling the swelling. In the neutron experiment, the void densities of the alloys were all about the same or greater than the densities of the pure nickel control. All materials, with the exception of the Ni-Si alloy, had mean void diameters similar to or greater than the pure nickel control. Therefore, in the Ni-Al, Ni-Ti and the Ni-Mo alloys, both nucleation and growth of voids were enhaced or unaffected. Only in the Ni-Si alloy, in which the void size was smaller than the pure nickel control, is it apparent that void growth was suppressed. In the ion irradiation, however, all alloys had lower void densities, so that suppression of void nucleation was occurfring. The mean void diameters for the Ni-Al, Ni-Ti and Ni-Mo alloys are all roughly the same as the pure nickel control, so that it does not seem that void growth was significantly affected.

The results of this experiment indicate that great care is necessary in using charged particle irradiation as a screening tool for assessing relative behaviors of alloys under neutron irradiation. Controlling mechansms for the properties of interest must be carefully studied to ensure that the same mechanisms will control the materials' behavior under both types of irradiation. If different mechanisms control in the two cases, faulty conclusions may be made regarding the effects of alloy modifications.

Acknowledgement

The authors wish to thank P. R. Malmberg for his assistance with the ion irradiations and D. I. Potter and P. R. Okamoto for helpful discussions on the effects of alloying additions on void formation.

Table 1 VOID AND DISLOCATION DATA FOR ION AND NEUTRON IRRADIATED NICKEL AND NICKEL ALLOYS

Temp. °C	Swelling %	Void Dens. $\#/cm^3$	Void Dia. A	Disl. Dens. $\#/cm^2$
Pure Ni				
675	4.1	2.3×10^{15}	308	1.4×10^{10}
625	3.3	4.2×10^{15}	237	2.0×10^{10}
525	2.7	1.9×10^{14}	610	2.7×10^{10}
455*	2.6	3.5×10^{14}	435	-
Ni 1% Al				
675	0.016	4.0×10^{12}	392	4.9×10^{9}
625	0.072	5.3×10^{13}	304	1.8×10^{10}
525	0.008	3.5×10^{13}	160	3.0×10^{10}
455*	3.0	4.2×10^{14}	488	-
Ni 1% Ti				
675	0.36	1.1×10^{14}	394	4.3×10^{10}
625	0.11	2.7×10^{14}	191	3.9×10^{10}
525	0.19	3.3×10^{14}	182	3.6×10^{10}
455*	4.9	2.8×10^{14}	621	-
Ni 1% Mo				
675	-	-	-	2.7×10^{9}
625	-	-	-	1.6×10^{9}
525	0.004	5.0×10^{12}	231	1.9×10^{10}
455	5.6	1.3×10^{15}	410	-
Ni 1% Si				
675	-	-	-	1.3×10^{9}
625	-	-	-	9.8×10^{9}
525	-	3.8×10^{14}	-	
455*	0.27	3.8×10^{14}	210	1.9×10^{10}

*Denotes data from the EBR-II fast reactor experiment.

B. The Microstructures of Titanium Scoping Alloys Following Low Fluence Neutron Irradiation at 450°C.

(J. A. Sprague and F. A. Smidt, Jr., Materials Science and Technology Division)

Introduction

Titanium alloys are among the several classes of materials currently being considered in the DOE Fusion Materials Program for fusion reactor first-wall and blanket applications. Although titanium alloys have been extensively studied, mostly in connection with aerospace applications, little is known about their response to neutron irradiation. The first step in gathering the necessary irradiation damage data on Ti-alloys for the fusion materials program is the investigation of a series of scoping alloys [24] which include the three major alloy types: near-alpha (also called super-alpha), alpha plus beta, and near-beta alloys. The first opportunity to examine these alloys after neutron irradiation has been provided by the stress-relaxation experiment previously reported by Nygren [25]. The microstructures of unstressed control specimens from this experiment have been studied by TEM in a joint effort among McDonnell-Douglas, Hanford Engineering Development Laboratory (HEDL), and NRL. In the NRL portion of the work, neutron-irradiated specimens of Ti-6Al-4V (beta-annealed), Ti-6242S, Ti-38-6-44, and Ti-15-333 were examined, and unirradiated specimens of beta-annealed Ti-6Al-4V and Ti-38-6-44 were further studied to aid the interpretation of irradiation-induced microstructural changes.

Experimental Procedures

The nominal compositions (weight percent) of the alloys examined in the present study were: Ti-6%Al-4%V; Ti-6%Al-2%Sn-4%Zr-2%Mo-0.09%Si; Ti-3%Al-8%V-6%Cr-4%Mo-4%Zr; and Ti-15%V-3%Cr-3%Al-3%Sn. The preirradiation heat treatments given to these alloys have been described by Davis, et. al. [24] and Nygren [25], but will be repeated here for ease of reference. The beta-anneal of Ti-6Al-4V was 1040°C/30 min plus A.C. plus 730°C/2 hour. The heat treatment of the Ti-6242S was 900°C/30 min plus A. C. plus 790°C/15 min plus A. C. The TI-38-6-44 and Ti-15-333 were both solution-treated and aged, the Ti-38-6-44 schedule being 815°C/30 min plus "rapid cool" plus 620°C/4 hour, and the Ti-15-333 schedule being 760°C/30 min plus A.C. plus 510°C/16 hours. The alloys were irradiated in EBR-II in the form of small beams, 1-mm thick. The irradiation conditions [26] were neutron fluence of 3.4×10^{21} n/cm^2, E>0.1 MeV, a damage level of 2.1 dpa, and an irradiation temperature of 450°C. Following irradiation, TEM specimens were prepared at HEDL by chemically milling the beams to thicknesses of approximately 0.2 mm in a solution of 65% HNO_3, 18% HF, and 17% H_2O at 0°C, and punching 3-mm disks [27]. The specimens were then shipped to NRL for final preparation and TEM examination. Final thinning was performed in a dual-jet electropolisher using a solution of 300 ml methyl alcohol, 175 ml n-butyl alcohol, and 15 ml perchloric acid at a temperature of $-60^{o}C$. The specimens were examined in a JEM-200A electron microscope operating at 200 kV.

Results and Discussions

Ti-6Al-4V, Beta-Annealed. The preirradiation microstructure of the beta-annealed Ti-6Al-4V, shown in Fig. 7(a), consisted primarily of acicular Widmanstatten alpha plates with interplatelet beta. The dark bands which appear between the alpha and beta phases in this micrograph are the interface phase, an fcc transition structure which forms during cooling from above the beta transition temperature [28]. After irradiation, as illustrated in Fig. 7(b), the basic Widmanstatten structure was retained, and dense internal damage structures developed in both the alpha and beta phases. Due to imaging difficulties, caused by the size and shape of the beta-phase plates, only the damage structure in the alpha phase was examined in any detail. The principal postirradiation features of the alpha-phase plates are illustrated in Fig. 8, a bright field-dark field series from one area of the specimen. The bright field micrograph, Fig. 8(a), imaged some large beta plates, dislocation loops, and some nearly linear features which appeared to be precipitates. The selected area diffraction of this area, Fig. 8(b), indicated a systematic $(10\bar{1}0)$ diffraction condition, with satellite spots and streaks surrounding the primary reflections. The primary $(10\bar{1}0)$ dark field image, Fig. 8(c), imaged the dislocation loops. The dark field micrograph formed with a satellite spot and streak, Fig. 8(d), imaged the large beta plates and a set of linear features with their long axes perpendicular to the streak.

When imaged under a number of beam directions and diffraction conditions, the linear features shown in Fig. 8 were identified as beta-phase precipitates, which agrees with identifications made by Sastry, et. al [29], and Powell [27] of similar features observed in mill-annealed and duplex-annealed Ti-6Al-4V from the same irradiation experiment. The beta precipitates were approximately planar in form with a habit plane close to $(11\bar{2}0)$ in the parent alpha grains. The orientation of the beta precipitates was the common Burgers orientation relationship [30] produced by the transformation of beta phase to alpha phase on cooling of Ti-6Al-4V below the beta transus temperature.

Three mechanisms were considered for the precipitation of beta phase in alpha grains of Ti-6Al-4V during irradiation:acceleration of the diffusion kinetics of vanadium, allowing a supersaturated alpha matrix to decompose at temperatures at which thermal diffusion would be too slow to cause observable precipitation; irradiation-induced segregation [31] of vanadium to point defect sinks, increasing the local concentration beyond the phase boundary; irradiation-induced alteration of the relative stability of alpha and beta phases at a given concentration [22]. Regarding

Fig. 7 — Microstructures of beta-annealed Ti-6Al-4V: (a) unirradiated, showing Widmanstatten structure of alpha plates separated by interplatelet beta; (b) irradiated, showing retention of Widmanstatten structure with addition of dense internal damage structures in alpha and beta phases

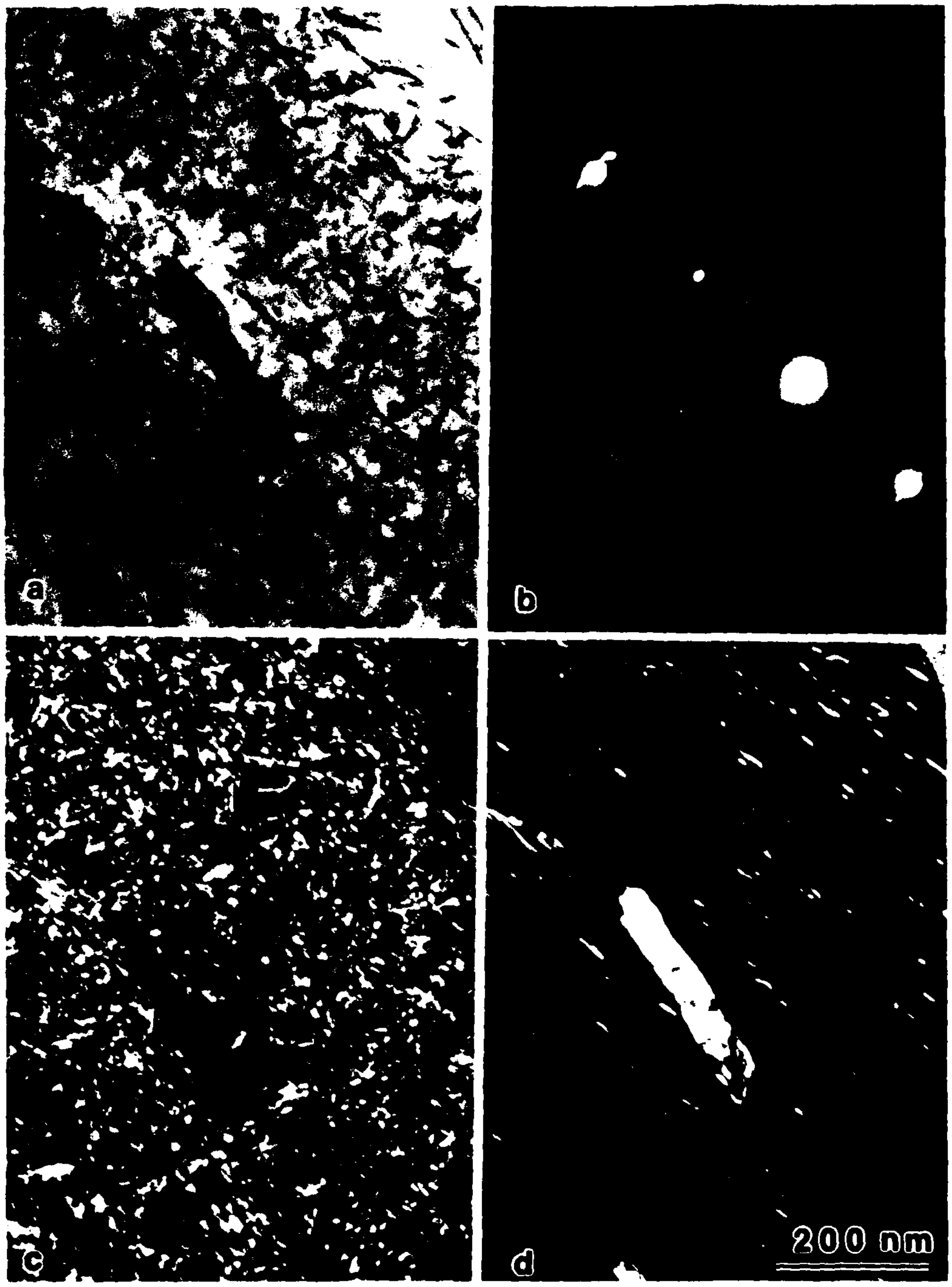

Fig. 8 — Internal damage structure in an alpha grain of irradiated Ti-6Al-4V: (a) bright field image, containing beta plate, dislocation loops and fine beta precipitates; (b) SAD of the area, in a systematic $(10\bar{1}0)_\alpha$ diffraction condition, with $(110)_\beta$ satellite spots and streaks; (c) dark field, $(10\bar{1}0)_\alpha$ reflection, showing dislocation loops; (d) dark field, $(110)_\beta$ reflection, showing large beta plate and fine beta precipitates

the possible existence of a supersaturated alpha phase, Rhodes and Paton [37], in their explanation of interface phase formation, note that the alpha-beta transformation occurs on cooling through the beta transus temperature by the growth of alpha plates into the beta matrix. If the cooling rate is sufficiently rapid, they state that the alpha phase will form at a critical vanadium content higher than the equilibrium content for this phase. Looking at the Ti-Al-V phase diagram, however, it is difficult to use this argument to explain the precipitation of beta phase at 450^{o}C. The diagrams published by Molchanova [33] indicate that the alpha phase in an alloy containing 6% Al should be stable at temperatures below 600^{o}C for up to 4% V, the total vanadium content of Ti-6Al-4V. The supersaturation of alpha plate with respect to vanadium concentration therefore does not seem likely.

Irradiation-induced segregation of vanadium, on the other hand, offers a possible explanation for the observed precipitation. Although the association of precipitates with point defect sinks could not be proven in the present study, Sastry et. al [29], reported that the precipitates in irradiated mill-annealed and duplex-annealed Ti-6Al-4V formed preferentially on dislocation lines and low-angle boundaries. For fcc crystals, it has been shown theoretically [31] that solute elements which trap self-interstitial atoms will segregate, and possible precipitate, at point defect sinks during irradiation. In nickel, the segregation and precipitation of silicon, which has a negative lattice misfit, has been observed by Auger analysis of ion-irradiated surfaces. Recognizing that the generalization of results from fcc nickel to hcp titanium, may not be valid, vanadium in alpha titanium has misfits of -3% on the a-axis and -6% on the c-axis [35] so vanadium segregation during irradiation might be expected. It should be noted, however, that aluminum in alpha-titanium has misfits of 0% on the c-axis (at low concentrations) and -6% on the a-axis, so that the situation in an alloy containing both elements could be complex. Segregation-induced precipitation of beta phase during irradiation, however, remains a distinct possibility.

The alteration of the alpha-beta phase boundary by irradiation-produced point defects cannot be ruled out as a mechanism for the observed precipitation, but the assessment of this possibility was well beyond the scope of the present study. There is a potential coupling mechanism for this type of phase stability effect, since the thermal alpha to beta transformation is accompanied by a volume contraction of approximately 5%, but a considerable modeling effort would be required to translate this information into a phase diagram. This question, therefore, was left open.

The pre-irradiation heat treatment of Ti-6242S produced a phase distribution consisting principally of equiaxed primary alpha with a small amount of transformed beta. As shown in Fig. 9(a), this general structure was not significantly affected by the irradiation. When imaged at higher magnification, as in Fig. 9(b), the transformed beta grains appeared to have a very fine and irregular platelet structure. The contrast from these

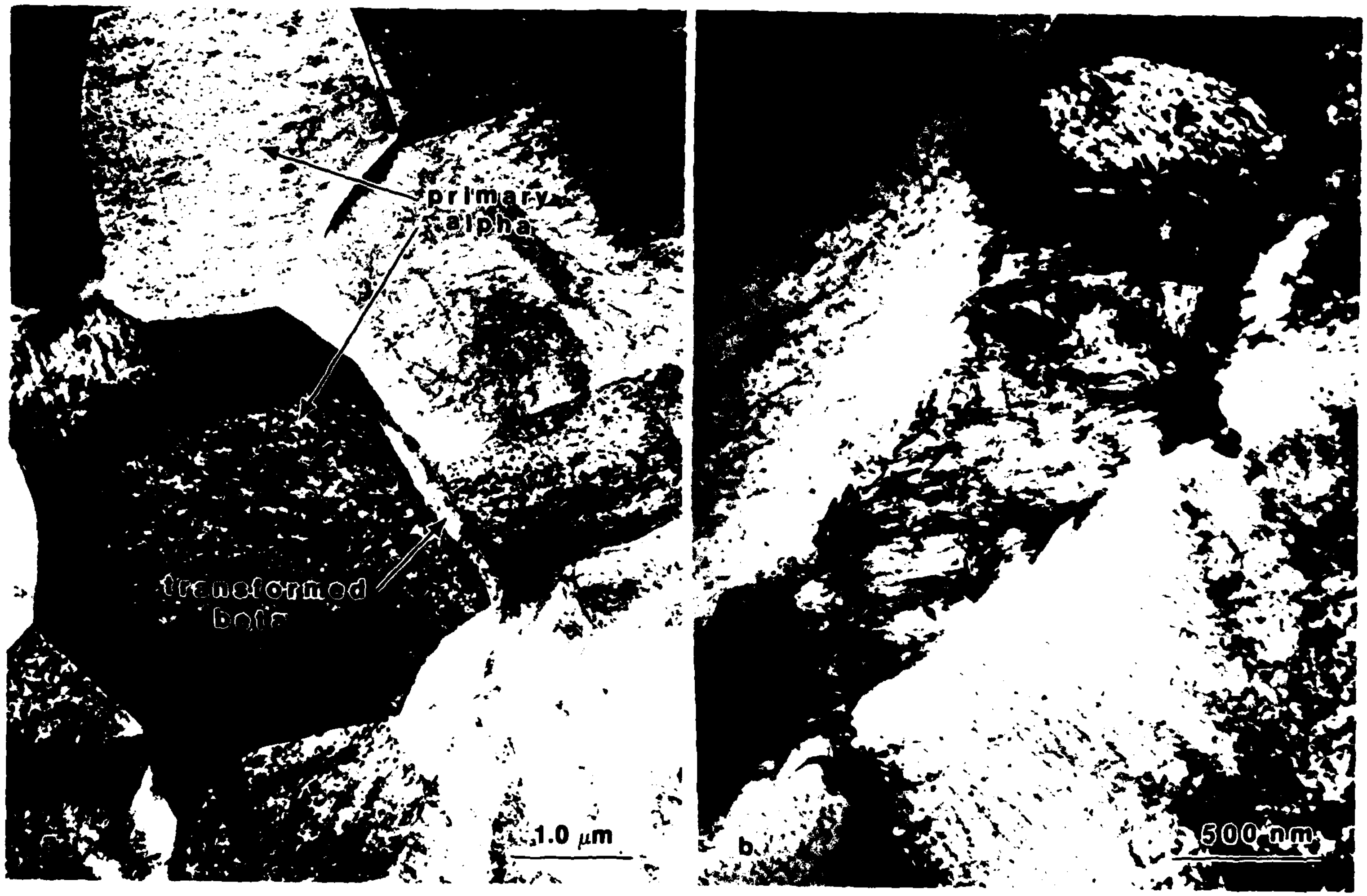

Fig. 9 — Phase distributions observed in neutron irradiated Ti-6242S: (a) general microstructure, showing equiaxed primary alpha and transformed beta grains; (b) internal structure of a transformed beta grain

dislocation loops, some of which appeared to be clustered together. While there was no evidence of precipitation in the diffraction contrast images of the alpha grains, the electron diffraction patterns of these grains contained weak reflections that could be identified as the alpha-2 phase, as shown in Fig. 10(b). This phase, which is $Ti_3(Al,Sn)$ with the DO_{19} hexagonal superlattice structure, would produce $[11\bar{2}0]$ reflections approximately halfway between the $[11\bar{2}0]$ reflections of the primary alpha, as seen in the figure. Attempts to image the alpha-2 in darkfield to check for association of this phase with the irradiation-induced dislocation loops were not successful, due to the weakness of the reflections. Although alpha -2 was not reported in the pre-irradiation microstructure of Ti-6242S [24], this may not be an irradiation-induced phase, since it is often observed in this class of alloys following normal heat treatment or long-term thermal aging.

Ti-38-6-44. The preirradiation microstructure of Ti-38-6-44, as discussed by Davis, et.al,[24], consisted of a beta matrix with large Type-2 alpha precipitates, shown in Fig. 11(a). Type-2, or "non-Burgers," alpha forms in many mestastable beta Ti-alloys, in which it appears to be the more stable form of the alpha phase, forming at higher aging temperatures and longer aging times from the initially-precipitated Type-1 (Burgers orientation) alpha.[36,37] After neutron irradiation at 450°C, the preirradiation alpha precipitate had grown somewhat, and additional fine-scale Type-2 alpha phase had precipitated, as shown in Fig. 11(b). The precipitation of additional alpha phase during irradiation was very likely the result of the normal thermally induced phase transformation, altered by irradiation-enhanced diffusion, since a larger volume fraction of alpha phase could be stable at 450° than at the aging temperature of 620°C.

The size of the fine Type-2 alpha precipitates observed after irradiation at 450°C was consistent with the precipitation reported by Rhodes and Paton [37] after aging this same alloy at 350 and 500°C. Their results suggest, moreover, that the precipitation during irradiation would both increase the yield strength and decrease the ductility of the alloy. For reference, Rhodes and Paton found that an 8-hour age at 450°C produced 160 nm Type-2 alpha precipitates, giving a yield strength of 1133 MPa and a plastic failure strain of 8.9%, while a 28-day age at 350°C produced 15 nm Type-2 precipitates, giving a failure stress of 1379 MPa and no plastic strain. The irradiation-enhanced phase transformation, therefore, could have some serious effects on mechanical properties of this alloy.

Ti-15-333. This alloy was investigated in the irradiated condition only, results being compared to the microstructure shown by Davis, et.al.[24] At low magnification, as seen in Fig. 12(a), the irradiated microstructure was similar to that observed for Ti-38-6-44, and the electron diffraction patterns indicated Type-2 alpha precipitates in a beta matrix. The precipitates in the Ti-15-333, however, were somewhat smaller than those

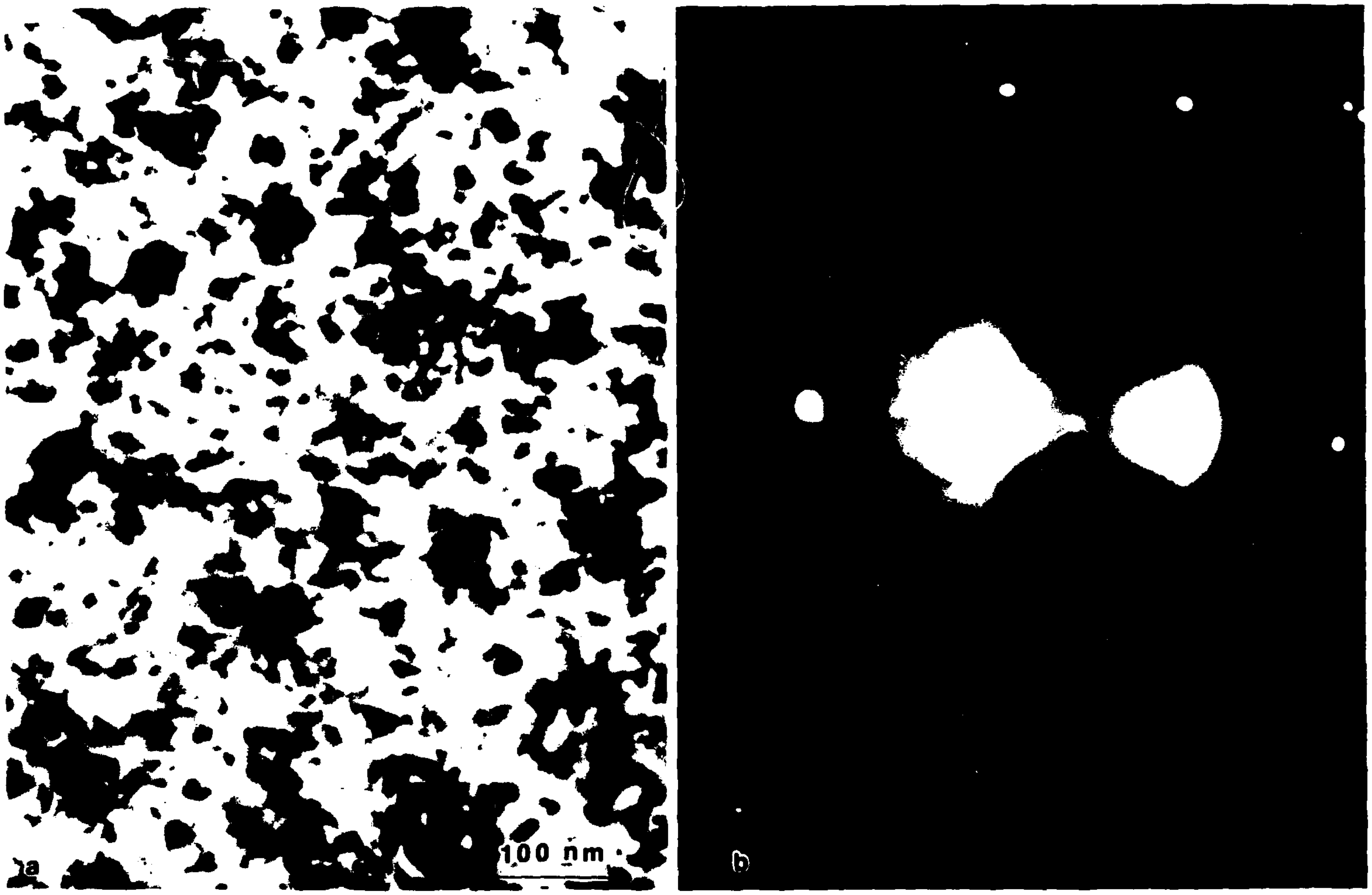

Fig. 10 — (a) Dislocation loops in primary alpha grain of neutron irradiated Ti-6242S; (b) selected-area diffraction pattern of (a) showing [11$\bar{2}$0] primary reflection and superlattice spots identified as [11$\bar{2}$0] $Ti_3(Al,Sn)$ reflections

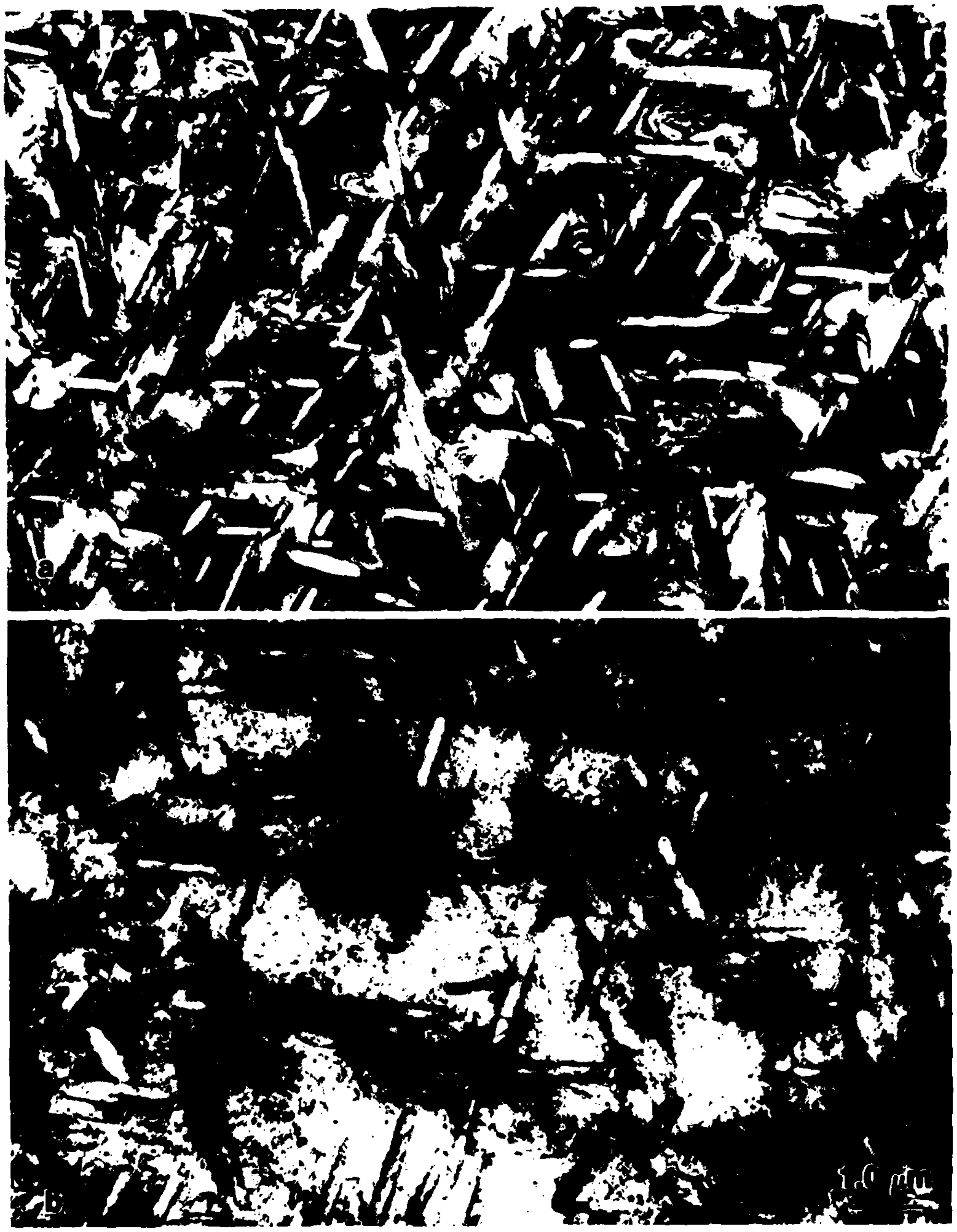

Fig. 11 — Microstructures of Ti-38-6-44: (a) unirradiated, with large Type-2 alpha precipitates in a beta matrix; (b) irradiated, exhibiting some growth of the preirradiation alpha precipitates and additional precipitation of fine Type-2 alpha phase

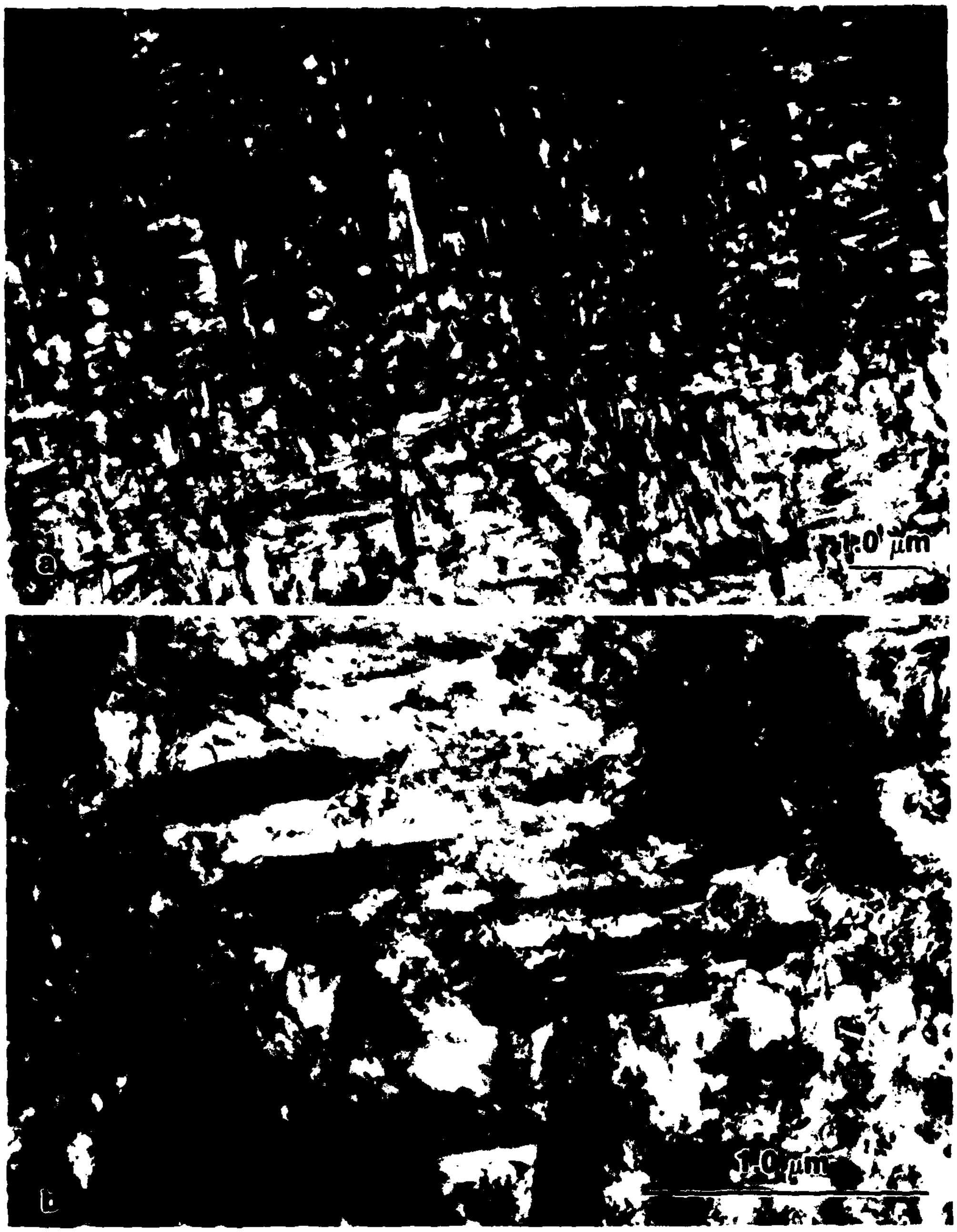

Fig. 12 — Microstructure of irradiated Ti-15-333: (a) low magnification, showing similar structure to Ti-38-6-44, but with smaller precipitates (see Fig. 11); (b) higher magnification, showing growth of alpha precipitates during irradiation (compare with Fig. 5.1.4.(b) of Davis, et al[1])

observed in Ti-38-6-44. Making a direct comparison of the irradiated microstructure at higher magnification, Fig. 12(b), with the preirradiation structure shown by Davis, et al.[24] [their Fig. 5.1.4(b)], the primary discernable effect of the irradiation was some growth of the preirradiation precipitates. The high precipitate density in the alloy made it impossible to detect any dislocation loops or other lattice defects. Since little information is readily available on structure-property relationships in Ti-15-333, it is difficult to speculate on the effect of the observed precipitate growth on mechanical properties. Qualitatively, one might expect less irradiation embrittlement of Ti-15-333 than of Ti-38-6-44 by looking at the microstructural changes, but more experiments would clearly be required to test this hypothesis.

Conclusions

Neutron irradiation of Ti-6Al-4V (beta-annealed), Ti-6242S, Ti-38-6-44, and Ti-15-333, to 2.1 dpa at 450°C produced the following microstructural changes:

Ti-6Al-4V - formation of dislocation loops and planar beta-phase precipitates in the alpha phase, as well as a damage structure in the beta phase which was not identified;

Ti-6242S - formation of dislocation loops in the alpha phase and an unidentified complex structure in the beta phase; some alpha-2 precipitation during either heat treatment or irradiation;

Ti-38-6-44 - some growth of the preirradiation Type-2 alpha precipitates and additional precipitation of fine Type-2 alpha in the beta matrix;

Ti-15-333 - growth of the preirradiation Type-2 alpha precipitates; but qualitatively less change in phase distribution than was observed in Ti-38-6-44.

To summarize these findings and the previous data generated in the joint McDonnell-Douglas, HEDL, and NRL study of the titanium scoping alloys in this irradiation experiment, the near-alpha alloys showed the greatest phase stability during neutron irradiation, with the alpha plus beta and near-beta classes both showing significant redistributions of the preirradiation phases. Although some speculation on the impact of these phase redistributions on mechanical properties is possible with these limited data, the more comprehensive test matrix planned for the DOE Fusion Materials Program will be required to assess the suitability of the alloys for the fusion reactor environment.

II. X-RAY DIFFRACTION MEASUREMENTS OF LOW-LEVEL RADIATION DAMAGE

(J. V. Gilfrich, D. B. Brown, J. W. Sandelin and L. S. Birks,
Radiation Technology Division)

Introduction

It is well known that two serious problems associated with the study of radiation damage in reactor materials are, (a) studies using neutrons are often prohibitively slow, and (b) simulation of neutron damage with heavy particles, though fast, produces results which are not easy to relate to neutron damage.

It is well documented in the literature that x-ray diffraction can be used to measure dislocation density in crystals and therefore should be useful in measuring early stages of radiation damage in reactor materials. We have proposed that alloy compositions which are resistant to void swelling might show different diffraction characteristics than alloys which are sensitive to void swelling. If such a correlation could be established then x-ray diffraction would be a promising tool for the study of reactor alloys because of the relatively low irradiation times required.

In an earlier CORES report [38] we (a) demonstrated that low levels of radiation damage are easily measured in single-crystal pure Cu, and (b) interpreted the measurements using a relatively simple model of the effect of dislocations on diffracted intensity.

Results

In this report we will describe three milestones in the development of this technique. (1) Our work on single crystal pure Cu has been extended to higher levels of damage from alpha particle bombardment. These results show that the diffracted intensity, which had risen significantly with low levels of irradiation, falls slightly with further irradiation. (2) We have developed a related experimental method using polycrystalline specimens. This is of importance because it is much easier to obtain adequate polycrystalline specimens than to obtain single crystals, and further, more information can be obtained using eight diffraction peaks instead of only one. (3) We have developed a more sophisticated model for the effect of radiation damage on the diffracted intensity. In this model the observed intensity increases are attributed to internal strain (lattice spacing variation) caused by the irradiation. The observed intensity decreases are attributed to dislocation loops.

In Fig. 13 we show our results for the diffracted intensity from the (111) planes of a pure Cu single crystal irradiated with 1.2-4 MeV alpha particles. It should be readily observable that two processes are oc-

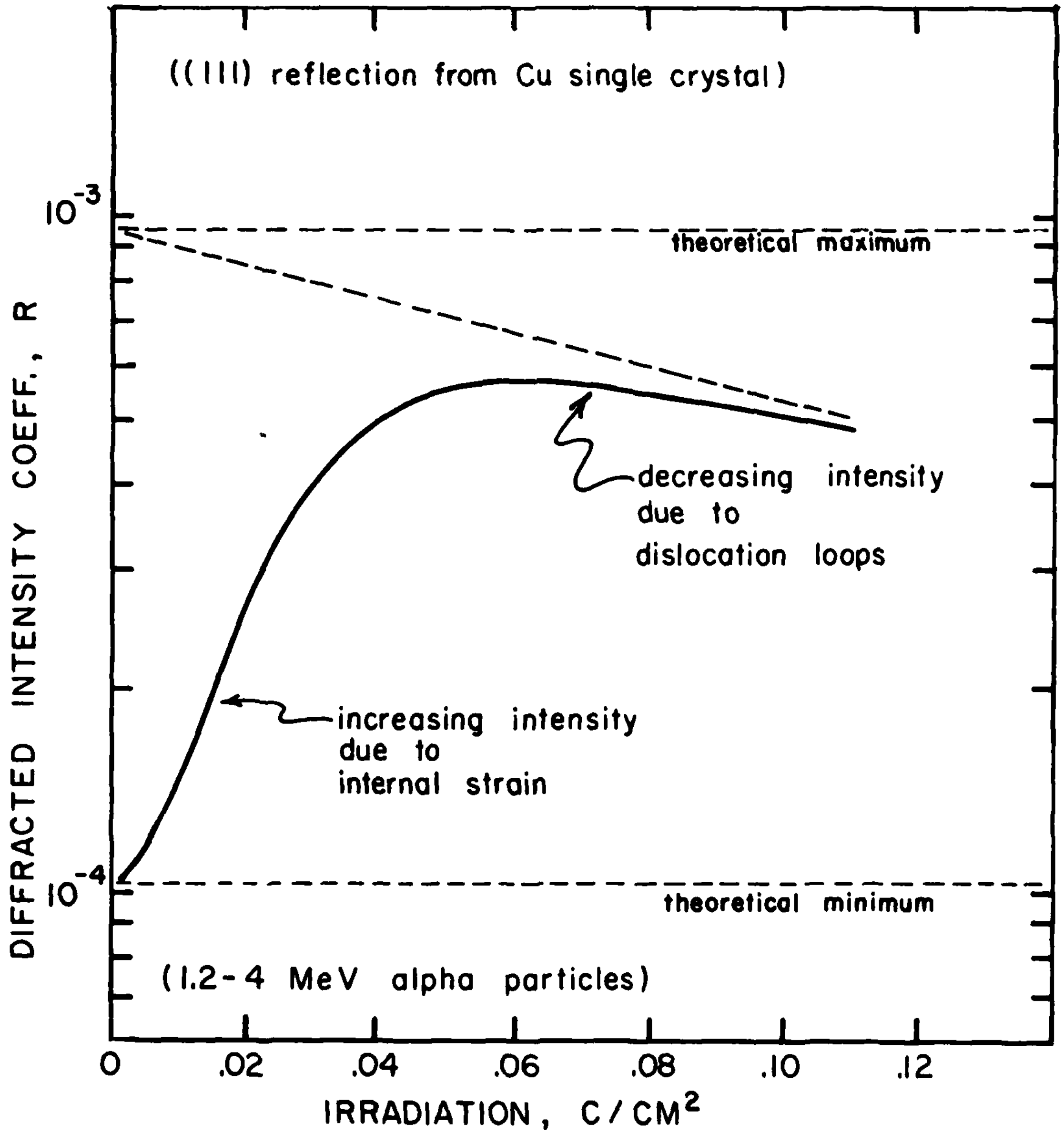

Fig. 13 — Diffracted intensity from the (111) planes of a pure Cu single crystal irradiated with 1.2-4 MeV alpha particles

curing. For the lower levels of irradiation the dominant process is the reduction of x-ray extinction caused by an increase in internal strain (lattice spacing variation). At the higher levels of irradiation x-ray extinction becomes negligible and therefore causes only slight further changes in intensity. In this region the dominant process is a reduction in intensity caused by dislocation loops.

In Fig. 14 we show the initial results from our new experiment using polycrystalline specimens. The results show the diffracted intensity from eight different diffracting planes of a pure Cu specimen. All intensities are normalized to the intensities from the specimen prior to any irradiation. Note that the result using the (111) diffracting planes is qualitatively the same as was observed using single crystal Cu. Note that some of the data, for example that using the (420) diffracting planes, do not show an initial rise. This is because, for these lines, x-ray extinction is not an important process and thus only the reduction in intensity due to dislocation loops is being seen. The small rise in the intensity of the (420) line at the highest irradiation levels is probably real. It is due to a diffuse scattering contribution caused by the dislocation loops.

We are beginning the necessary computer programming to extract quantitative estimates of three parameters relevant to the polycrystalline specimens. The analysis is a four step process:

(1) Fit theory to data from high index diffracting planes such as the (420) to obtain $\rho_{loop}\, r^3_{loop}$ as a function of the degree of irradiation. The parameter ρ_{loop} is the dislocation loop density, while r_{loop} is the dislocation loop radius.

(2) Using the above data, correct data from low index diffracting planes such as the (111) to eliminate the effect of dislocation loops.

(3) Extrapolate such corrected curves to their asymptotic values at high irradiation levels. These asymptotic limits should represent the theoretical maximum intensity and can be used to infer ρ_{line}, the concentration of dislocation lines which exist in the specimen prior to irradiation.

(4) Using all of the above information, fit theory to data from low index planes to obtain the lattice strain, $\Delta d/d$, as a function of irradiation.

We have begun a similar sequence of irradiation and analysis on Al and Be alloys of Cu. Alloys containing 1, 2.5, 5, and 9% Al have been given a first irradiation. Preliminary data suggests that 1% Al reduces both the strain and the dislocation loops in alpha particle irradiated Cu. Initial attempts have also been made to determine the availability of Al-Cu single crystals, so that the parallel experiment can be carried on.

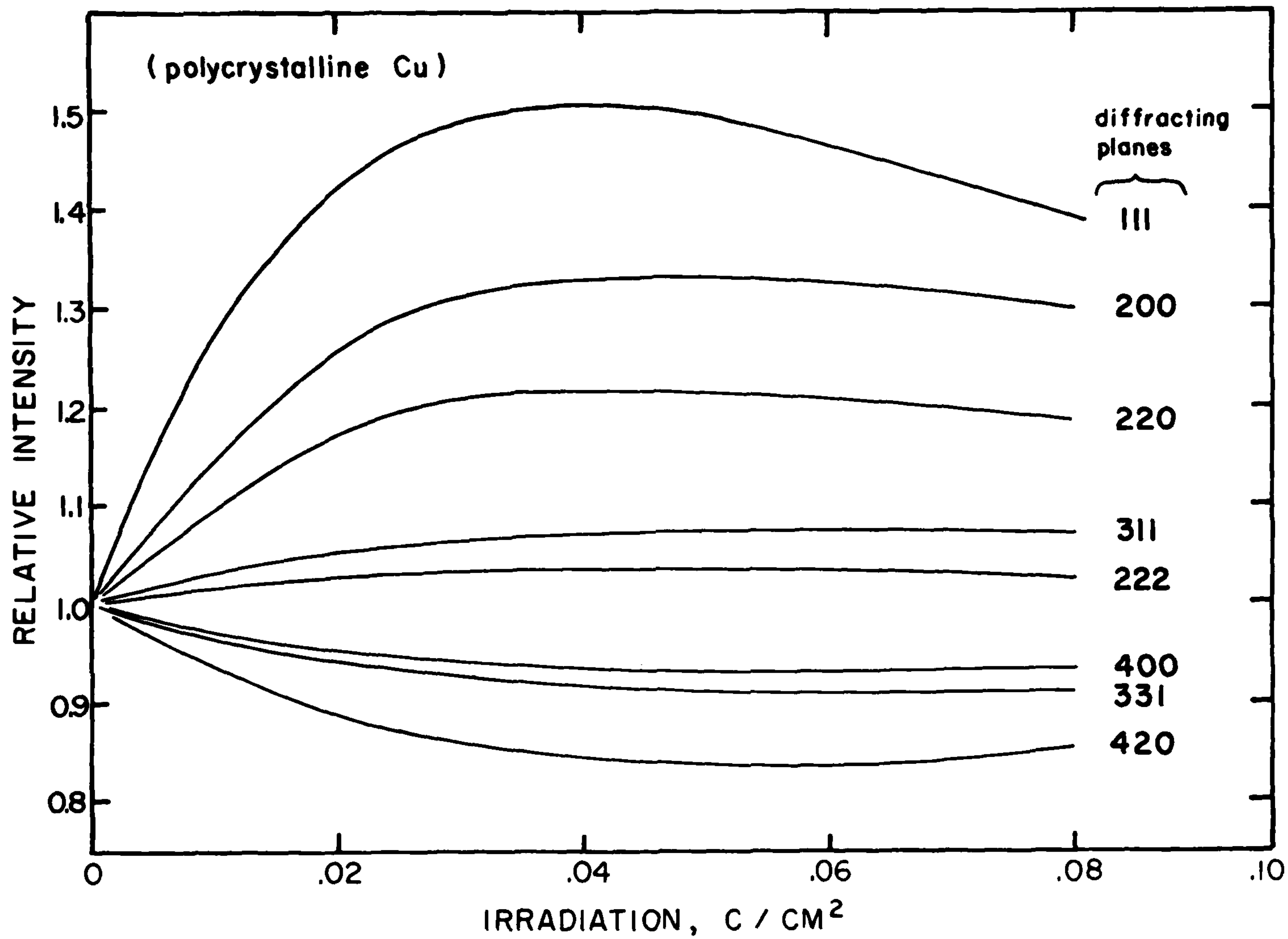

Fig. 14 — Diffracted intensities from polycrystalline specimens irradiated with 1.2-4 MeV alpha particles

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